Time-Resolved Ionisation Studies of the High Power Impulse Magnetron Discharge in Mixed Argon and Nitrogen Atmosphere

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Introduction

High power impulse magnetron sputtering (HIPIMS) is a new method for physical vapour deposition (PVD) based on magnetron sputtering [1,2]. It utilises transient impulse (short pulse) glow discharges with very high power and current density (up to 3 kWcm^{-2} and 4 Acm^{-2} respectively at a duty cycle of <5%). Under these conditions the plasma density near the target increases sufficiently to ionise a significant proportion of the sputtered metal ions [2,3] thus creating a high-efficiency metal ion source. The discharge has been up scaled successfully [4] and has found a number of applications. One example is substrate pre-treatment under HIPIMS which benefits from droplet-free metal ion bombardment that provides a highly clean coating-substrate interface where local epitaxial growth is maintained [5]. This in turn enhances the coating adhesion. Another example are nitride [6] thin films growth by HIPIMS. CrN films grown by HIPIMS have a high density of the microstructure and demonstrate superior performance in corrosion and wear environments [6,7].

The HIPIMS discharge is known to develop in two phases within each power pulse. Fast time-resolved optical emission spectroscopy (OES) studies have demonstrated a transition from gas phase to metal phase [2,8]. OES has also shown the existence of high proportions of ions and the presence of doubly-charged species [2]. Mass-spectroscopy results in inert gas atmosphere have indicated high metal ion content and high ion energy [3].

Although nitride films grown by HIPIMS in reactive atmosphere have demonstrated a superior performance which has been linked to the high content of N_2^{1+} observed in OES studies [6], however, quantitative characterisation of the discharge in reactive conditions has not been published to date.

Energy-resolved mass spectroscopy is used to characterise quantitatively the composition, energy and time evolution of the HIPIMS plasma in reactive atmosphere. The results are compared in relation with conventional direct current (DC) and mid-frequency (MF) pulsed DC sputtering at the same average power and composition of the atmosphere.

Experiment

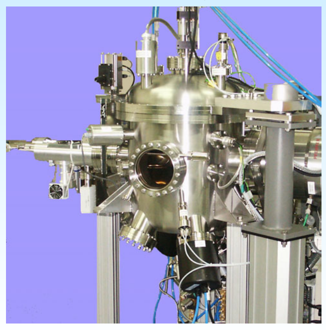
Equipment:

- Ultra high vacuum (UHV) chamber, base pressure of < 10^{-9} mbar.
- One magnetron (Torus™, Kurt J Lesker) diameter of ø75 mm Ti target
- HIPIMS Power Supply from Advanced Converters AC Sp.z o.o. Poland

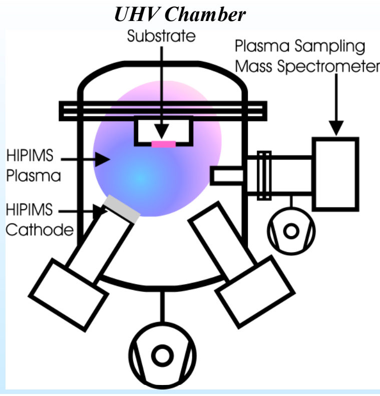
Energy-resolved mass spectroscopy at substrate position:

- PSM003, Hiden Analytical Ltd.
- Distance to target = 120 mm; Angle of 58° with respect to target normal.
- Plasma sampled through a ø200 µm grounded orifice, acceptance angle of 5°
- Ion collection gated with a TTL output from power supply
- Measure a 70 µs window centred at the peak of the 150 µs pulse.

Photo of UHV Chamber – Kurt J Lesker, Ltd.



Schematic Cross-section of UHV Chamber



NEW: Quantitative Plasma Analysis of HIPIMS in *REACTIVE ATMOSPHERE*

Comparison:

HIPIMS, DC and mid-frequency pulsed DC

- Identical average power = 200 W
- Pulsed DC frequency = 20 kHz
- HIPIMS Discharge:
  - Peak power = 20 kW,
  - Peak current = 50 A
  - Pulse frequency = 100 Hz.
  - Discharge voltage:
    - -550 V for inert gas atmosphere
    - -480 V for reactive gas mixture.

Inert atmosphere: Ar

Ar pressure = 2.5×10^{-3} mbar.

Reactive atmosphere: Ar + N_2

PAr = 2.5×10^{-3} mbar,

$PN_2 = 2\times10^{-4}$ mbar

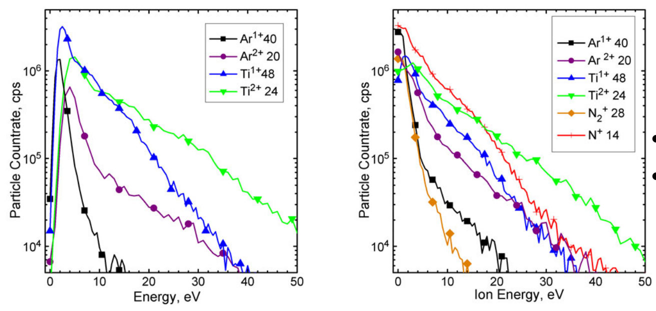
Results

The time-averaged ion distribution function (IEDF) for ions generated by different method of sputtering in inert and reactive atmospheres are shown below:

Ion energy distribution functions (IEDF)

HIPIMS of Ti

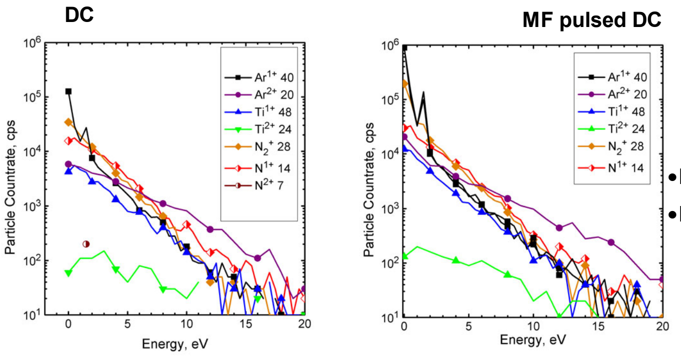
inert atmosphere: Ar — reactive atmosphere: Ar + N_2



- High maximum energies
- High content of N+

DC & MF pulsed DC Sputtering in Reactive Sputtering

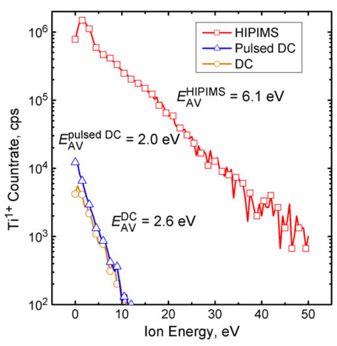
DC — MF pulsed DC



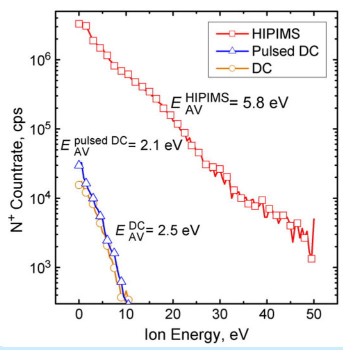
- Low maximum energies
- Lower content of N+

IEDFs of the film forming species – Comparison HIPIMS, DC, pulsed DC

Ti^{1+}



N^{+}



- Three-fold increase in average energy for HIPIMS in comparison to DC and Pulsed DC

Ions were sampled with an energy-mass spectrometer PSM003 (Hiden Analytical Ltd)

Ion content, %

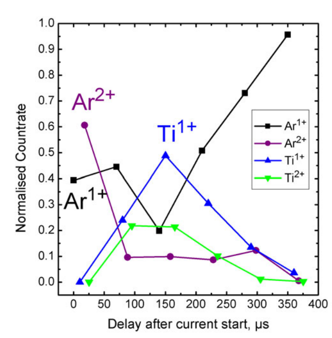
Technology	Gas composition	Ar^{1+}	Ar^{2+}	Ti^{1+}	Ti^{2+}	N_2^{+}	N^{+}
HIPIMS	Ar	34	5	**46**	**15**	-	-
Pulse DC	Ar	**78**	9	13	0.2	-	-
DC	Ar	**82**	8	11	0.2	-	-
HIPIMS	Ar + N_2	19	5	**19**	**10**	7	**40**
Pulse DC	Ar + N_2	**52**	4	4	0.07	**31**	9
DC	Ar + N_2	**65**	3	3	0.03	**23**	6

Time resolved measurements

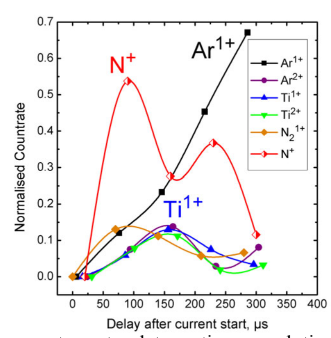
Initially the discharge consists of singly and doubly ionised Ar. After 70 µs, the Ti^{1+} and Ti^{2+} ions comprise the majority of ion flux – some 70% of the total. This phase coincides with the peak in discharge current and extends over 100 µs. At this time the Ar^{1+} and Ar^{2+} content diminish quickly due to the intense production of metal ions and the rarefaction of the gas by the high power dissipated in the pulse.

At the end of the pulse at 250 µs the power is switched off and the metal signal decays as the voltage is insufficient to sustain the sputtering process. The plasma comprises mainly Ar ions generated by remnant electrons that ionise the gas present in the chamber and electron waves generated during the power pulse and reflected by the chamber walls.

These observations fit well with published OES results measured for Cr, showing the transition from a gas discharge to a metal discharge phase [2]. However the present results illustrate the point in a quantitative manner and demonstrate the relatively high content of metal ions generated by HIPIMS.



Mass-spectrometer data on time - evolution of a HIPIMS of Ti discharge in an inert gas (Ar) atmosphere



Mass-spectrometer data on time - evolution of a HIPIMS of Ti discharge in a reactive gas (Ar/N_2) atmosphere

The energy-resolved mass spectrometer was synchronised with the TTL output from the HIPIMS power supply, and was adjusted to measure for a 70 µs window centred at the peak of the 150 µs pulse.

Conclusions

The composition and energy of the ion flux in HIPIMS, pulsed DC and DC discharges was investigated quantitatively in inert (Ar) and reactive gas (Ar/N_2) atmospheres. The ion flux in HIPIMS was found to contain factor of five more metal ions and factor of four more reactive nitrogen ions compared to conventional sputtering techniques. The film forming species of Ti and N were present in ionised form in HIPIMS thus allowing high control over the deposition conditions. Ion energy distribution functions revealed factor of three higher average energies for the HIPIMS case due to the ionisation of reflected fast neutrals of both gas and metal.

The time evolution of a HIPIMS pulse in reactive gas atmosphere was similar to inert gas atmosphere. Distinct stages of gas-dominated and metal-dominated plasma were detected. The peak metal ion content was 60% for the inert gas case and 30% for the reactive gas case (Table 1).

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